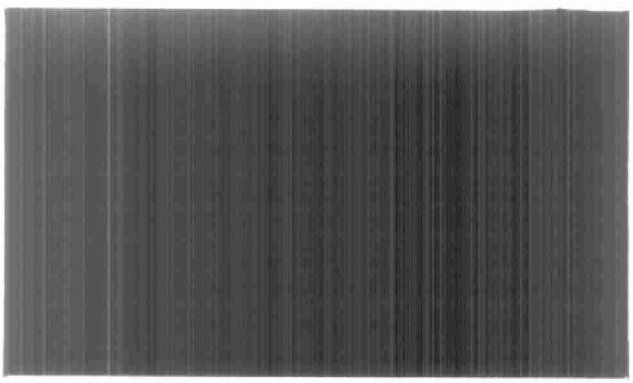
...ter management in Ontario

Ontario
Water Resources
Commission

Great Lakes
Water Quality
Surveys Program



# SAMPLING AND ANALYTICAL

# TECHNIQUES FOR

# GREAT LAKES SURVEYS

June, 1970

TABLE OF CONTENTS

# I USE OF CHEMICAL AND BACTERIOLOGICAL SAMPLERS

## 1. KEMMERER SAMPLER

### (A) Description and Purpose of the Kemmerer Sampler

The Kemmerer sampler is a brass sampler with a 3 litre capacity. Its purpose is to bring an uncontaminated water sample from a desired depth to the surface. The sampler is lowered on the oceanographic wire in the open position, thus flushing itself during the lowering. When the sampler reaches the predetermined depth, a brass messenger is released to trip the Kemmerer sampler. The sampler then closes entrapping a water sample from the desired depth.

### (B) Preparing the Kemmerer Bottle for Operation

Before a Kemmerer sampler is used, it should be checked carefully for proper operation of its parts. The tripping springs at the top of the sampler should be checked for stiffness, if they are not stiff the sampler could close automatically as the sampler is lowered. Check to make sure that the two ends of the sampler close properly and that the rubber is not cracked or grooved.

### (C) Placing the Kemmerer Sampler on the Wire and Loading

(1) Put the Kemmerer sampler messenger on the wire.

(2) Put the Kemmerer sampler on the wire by putting the wire through the central core. Make sure that the firing mechanism is up toward the messenger.

(3) Shackle a lead weight to the wire.

(4) Place a bacti sampler about one foot above the Kemmerer sampler (see section on bacti sampler).

Loading

(1) Attach the Kemmerer sampler messenger to the bacti sampler with the looped wire of the messenger.

(2) Grasp the rubber top of the Kemmerer sampler and pull upwards until the loading mechanism is engaged.

(3) The sampler is now ready for use.

(D) Lowering the Kemmerer Sampler

Lower the sampler slowly until the air is expelled and then gradually increase the speed until the desired depth is reached. Slow the descent of the sampler before the desired depth is reached and then stop. Do not stop the sampler suddenly or it may fire automatically. When the proper depth has been reached trip the samplers by a messenger from deck level. Now bring the sampler to the surface.

## 2. NANSEN BOTTLES

(A) Description and Purpose of Nansen Bottles

The Nansen bottle is a metal reversing water sampler with a 2.0 liter capacity. Its purpose is to bring an uncontaminated water

sample from a desired depth to the surface. It is fitted with a tapered plug valve at either end and is lowered on the oceanographic wire in the open position thus flushing itself during the lowering. When the bottle reaches the predetermined depth, a brass messenger is released to trip the Nansen bottle. The messenger serves to disconnect the top of the bottle from the wire; the bottle then reverses, making a $180^{o}$ arc with the wire. The plug valves close when reversal occurs, entrapping a water sample from the desired depth, and a second messenger which is attached to the first Nansen bottle is released which in turn effects the reversal of a lower bottle, and so on when they are attached in series.

Each Nansen bottle is fitted with a frame to hold reversing thermometers. These thermometers are inserted in slotted brass tubes attached to the frame to permit easy reading of the scale. One end of each tube is perforated to permit water circulation so that the thermometers may come to temperature more rapidly. The ends of the tubes contain coil springs and rubber pads to hold the thermometers securely and provide a certain amount of protection against shock. The thermometers are always inserted into the tubes in such a manner that the large mercury reservoir is in the end of the tube having the small perforations.

(B) Preparing the Nansen Bottle for Operation

Before a Nansen bottle is used, it should be checked carefully for proper operation of parts. The valves should be lubricated with a silicone stopcock grease to ensure smooth movement. All moving parts should be lubricated to give free action. Springs and pins of the messenger and bottle-releasing mechanisms should be tested for proper functioning. The action of the air vent screw and the condition of the washer should be checked. The vent holes must not be clogged. The drain petcock valve should turn smoothly. After the bottles have been checked and are in operating condition, they are arranged in the rack in the order of use.

(C) Placing the Nansen Bottle on the Wire

After the lead weight has been shackled to the wire and lowered over the side, it usually is run 1 meter below the waterline and the winch is stopped. This holds thelead clear of the hull and the wire steady against the roll of the ship. The depth gauge disc is zeroed and the Nansen bottles are fastened to the wire in the loaded state with the valves open and the thermometer in the reversed position. The bottle is then clamped securely to the wire by tightening the wing nut. Depress the messenger trigger of the tripping assembly on the upper end of the bottle and fasten

it to the wire. Make sure that the messenger trigger returns to the "up" position and the pin holding the bottle to the wire has returned completely to the closed position. Next check to ensure that the drain petcock and the air vent are both closed. Check the reversing thermometer to ensure that the mercury has drained out of the bulb into the reservoir. Tapping the glass with the fingers usually will release the mercury from the bulb and permit it to drain. It is most important to see the mercury has drained before lowering the Nansen bottle. To all bottles except the first bottle placed on the wire, a messenger must be attached. Each messenger has a length of wire about 6 or 8 inches long, with a small eye in the outer end. This eye is attached to the Nansen bottle by inserting it into the small slot in the underside of the clamp assembly. Depress the release arm, insert the eye, and release the arm. Make sure the messenger release pin has seated itself through the eye. Attach the messenger to the wire below the bottle. A bacti sampler is normally attached above each Nansen bottle (see section on bacti sampler). The bottle is now ready for lowering.

(D) Lowering the Nansen Bottle

Lower the Nansen bottle slowly until it has entered the water. After the bottle has reached a couple of meters below the surface gradually increase the speed of the winch to its normal lowering speed.

After the last bottle has been lowered to its indicated depth, plus the distance from the platform to the surface, the winch is stopped. Leave the Nansen bottle at the predetermined depth for 5 minutes, then trip the first Nansen bottle by means of a messenger.

(E) Bringing the Nansen Bottle In

After sufficient time has been allowed for the lowest bottle to be tripped, bring up the bottles. Remove the messenger from the wire above the bottle. Unclamp the Nansen bottle from the wire and return it to the rack, being very careful to keep the bottle always in a vertical position with the clamp assembly at the top. This will prevent accidental reversing of the thermometers.

When all the Nansen bottles have been returned to the rack, the water samples are drawn. After all water samples have been drawn, drain the excess water from the Nansen bottle.

(F) Storing the Nansen Bottles

When storing Nansen bottles overnight, keep them in the reversed position, i.e. the wing clamp is at the bottom end of the bottle. This is necessary only when the reversing thermometer is attached.

## 3. THE BACTERIOLOGICAL SAMPLER (Bacti Sampler)

### (A) Description and Purpose of the Bacti Sampler

The Bacti sampler's purpose is to bring a water sample in a sterilized bulb from predetermined depth to the surface. The sampler is loaded with a new sterilized bulb on each lowering.

Each bulb is fitted with a metal plug and when the proper depth is reached the sampler is tripped by means of a messenger and the plug is pulled from the bulb, thus it fills with water.

### (B) Preparing the Bacti Sampler for Operation

Before the bacti sampler is used, it should be checked carefully for the proper operation of its parts. The springs on the sampler should be checked to make sure that they are operating properly. The springing action should remain strong. Make sure that the two chains are attached firmly to each arm and that the plunger is functional.

### (C) Placing the Bacti Sampler on the Wire

The bacti sampler is usually placed above the Kemmerer sampler and is positioned about one foot above the samplers. To place the bacti sampler on the wire, check to make sure that the firing plunger is facing up then depress the plunger on the side of the sampler and insert the wire in the groove in the back of the sampler, then release the plunger. Check and make sure that the wire is held in place after

the plunger is released. Next attach the wing clamp to the wire and make sure that it is tight, because this clamp ensures that the sampler will not slide down the wire.

(D) Loading the Bacti Sampler

Attach a sterilized bacteriological bulb to the circular clamp on the loose arm. Before attaching, however, loosen the plug slightly or the plug may not come out when the sampler is tripped. Attach the bulb so that the bulb is flush with the surface of the clamp and so that the metal plug is facing the stationary arm of the sampler. Then depress the plunger on the top of the sampler and attach the chain from the loose arm to the pin which was moved by depression of the plunger. Now attach the messenger which is used to trip the next down line sampler by means of the looped wire from the messenger into the same position as the chain was attached; now release the plunger and make sure that it comes all the way up in order to hold the chain and messenger. Finally, just before sending the sampler down attach the chain on the stationary arm to the plug in the bacteriological bulb.

(E) Sealing the Bacteriological Bulb

After the bacteriological bulb has been retrieved immediately seal the bacteriological sample with a sterilized glass rod. The glass rod comes in a cellophane package. To take the glass rod out of the cellophane so that it will not be contaminated by handling grasp one end of

the rod in the left hand and tear open the other end of the cellophane bag. Then grasp one-half of the rod in the right hand and pull it out of the cellophane and then insert the glass rod up to half-way into the bacteriological bulb, then attach a label to the bulb and give the following information: sample number and date.

(F) Bacti Sampling with Nansen Bottles

A bacti sampler is attached securely to the barrel of each Nansen bottle. The reversing action of the Nansen bottle fires the bacti sampler and a bacti sample is collected as mentioned in the previous section.

4. SAMPLING DEPTHS

The following table gives a list of standard depths at which samples are to be taken during monitor surveys of the following lakes:

| 1. Body of Water | Sampling Depths |
|---|---|
| Lake Ontario | (1) 1.5 m from the surface;<br>(2) 10.0 metres from the surface, where possible; |

| | Body of Water | | Sampling Depths |
|---|---|---|---|
| | | * (3) | (Total depth minus 3 metres), i.e. 3 metres from the bottom whenever the total depth ≥17 metres. |
| 2. | Lake Erie | (1) | 1.5 m from the surface; |
| | | (2) | 7.0 metres from the surface, where possible; |
| | | * (3) | (Total depth minus 3 metres), i.e. 3 metres from the bottom whenever the total depth ≥14 metres. |
| 3. | Lake St. Clair | (1) | 1.0 metres from the surface; |
| | | (2) | 3.0 metres from the surface, where possible. |
| 4. | Lake Superior<br>Lake Huron | (1) | 1.5 m from the surface; |
| | | (2) | 10.0 metres from the surface, where possible. |
| | North Channel<br>Georgian Bay | * (3) | (Total depth minus 9 metres), i.e. 9 metres from the bottom whenever 100 metres<br>OR<br>Total depth minus 3 whenever depth between 17 and 100 metres. |

* The following factors must be taken into account when deciding on a depth for a bottom sample:

(1) Rate of drift

(2) Time on Station

(3) Contour of the bottom

(4) Size of swell

(5) Wire stretch

(6) Wire angle

These factors should be taken into account when deciding on the depth for the lowest sample so that no equipment is broken or lost by hitting the bottom. If the crew chief feels that the bottom sample cannot be taken at the depths mentioned in the previous table, the crew chief may at his discretion, change the bottom sampling depth.

# II PHYSICAL

1. TEMPERATURE

Temperature measurements can be taken with a regular laboratory type Mercury thermometer, approximately 12" long and having a range of $0^{o}$ - $30^{o}$C. This type of thermometer is generally employed for surface temperature measurements, whereas in situ temperature at depth requires the use of a reversing thermometer or sometimes a bathythermograph. The latter is generally used for determining temperature profiles but can also provide a rough indication of temperature at depth.

(a) The Mercury Thermometer

(i) To take surface temperatures, the mercury thermometer should be immersed through the top of the Kemmerer sampler as soon as the sample is retrieved.

(ii) There should be a minimum of delay between sample retrieval and the measurement of its temperature.

(iii) The temperature should be read while the thermometer is immersed in the water sample.

(iv) Read the thermometer to the nearest $0.2^{o}$C. Since the thermometer is generally calibrated in whole degrees, fractional parts of a degree are estimated.

(b) The Reversing Thermometer

The reversing thermometer is generally used at all depths other than the surface.

How to Use the Reversing Thermometer

The reversing thermometer is attached to the outer casing of a Nansen bottle. The bottle is sent down to a predetermined depth and left at this depth for 5 minutes minimum. The Nansen bottle is tripped by means of a messenger. The thermometer should not be reversed again until the temperature has been read. The reversing thermometer is generally read to the nearest $0.1^{o}C$ and the temperature recorded.

When higher accuracy is required for temperature readings, the Culbertson circular slide rule is to be used to calculate a correction factor so that temperatures are recorded to the nearest $.02^{o}C$.

To use the Culbertson slide rule, three values must be known:

(i) T' - reading of the reversing thermometer to the nearest $.02^{o}C$.

(ii) t - reading of the auxiliary thermometer which accompanies each reversing thermometer (that is, the ambient temperature)

(iii) Vo - a constant for the individual thermometer, representing in degrees the volume of mercury

below the $0^{o}C$ mark when the thermometer is in the reversed position at $0^{o}C$.

The correction factor is calculated by setting the two arms of the slide rule to the two temperature measurements on the linear scale on the periphery of the blue-black-white spirally stripped scales. This scale is labelled T'-t or Tw-t. The long arm is set to the larger of the two temperatures and the short arm set to the lower of the two temperature values.

The coupled movement of the short and long arm is affected by means of the long arm. Rotate the long arm until the short arm is set at $0^{o}C$ on the peripheral scale.

Add $V_o$ + T' and locate this position on the scale of the large arm. Below this position read on the spiral curves the correction factor, these values are all less than $1^{o}C$.

If the air temperature is warmer than the water temperature, the correction factor is subtracted from the temperature of the reversing thermometer. If the water temperature is warmer than the air temperature, the correction factor is added to the temperature of the reversing thermometer.

(c) Bathythermograph

Equipment

- Bathythermograph (B.T.)

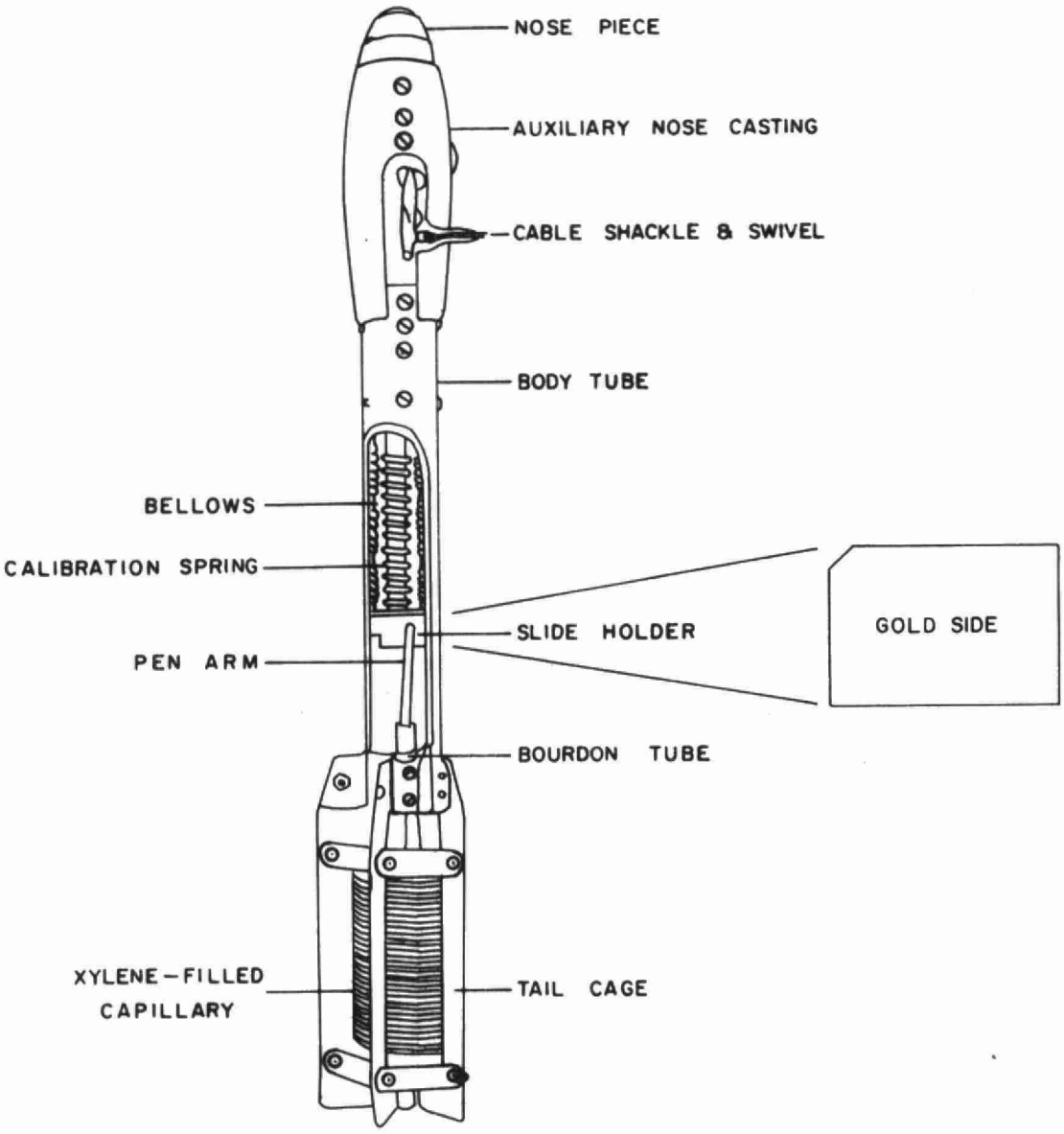
- B. T. - slides

- B. T. - slide reader

Fasten the B. T. to the wire with a screw type shackle and tighten the screw with pliers. Insert the slide into its holder in the body of the B. T., placing the edge of the slide with the bevelled corner in first. Make certain that the gold film of the slide is toward the stylus. Push the slide all the way in against the stop pin. It is important that the slide is fully in, otherwise the temperature will be recorded too low. Occasionally, check the grooves of the slide holder to make sure they are clean, free of glass chips, and that the spring holds the slide firmly against the opposite groove. When the slide is fully inserted, pull the protection sleeve back over the opening.

Lower the B. T. over the side of the vessel and leave it in the surface waters for 15-20 seconds to allow the thermal element to come to the temperature of the surface. Check total water depth on the fathometer (depth recorder) before lowering the B. T., as it is desirable that the B.T. should not be lowered closer than several meters from the bottom. The B.T. should be lowered quickly and then immediately raised at the same rate.

Exercise caution when the B.T. nears the surface to ensure that it does not hit the side of the vessel. As soon as the B.T. is onboard, move the sleeve forward toward the nose to lift the stylus




Bathythermograph

FIGURE 1

off the slide. Slack off the wire, place the B.T. on the deck. Take out the slide by the edges and carefully examine it to make sure that a suitable trace has been obtained. With a sharp pencil, write the following information in an open corner on the slide:

(1) Sample number
(2) Date
(3) B.T. serial number
(4) Surface temperature

Use the B.T. slide reader to read temperature vs depth. The B. T. slide reader is specifically built for each B.T. and may not be exchanged.

Important

(1) Do not leave the B.T. in the sun, as the fine capillary tubing will be ruined if the temperature exceeds 105°F.

(2) The sleeve cover of the B.T. should be left open when storing the B.T.

(3) Do not touch the face of the B.T. slide with fingers.

2. pH

(a) Equipment

- pH meter
- standard buffer solutions pH 4, 7 and 9

- beaker
- distilled water

(b) Standardization of the pH Meter for Measurements of Lake Water

When standardizing the pH meter, use buffers of pH 7 or 9. The choice of which particular buffer to use is based on the pH range of the water to be tested. Standardize the pH meter at pH of 7. Read the pH of the lake water and if the pH of the water is 8.6 or less, leave the pH meter standardized at the buffer of pH 7. However, if the lake water has a pH 8.7 or above, restandardize the meter with buffer of pH 9. Check the individual manual for the meter for the method of standardization.

(c) Standardization of the pH Meter for Alkalinity Use Buffer pH 4

(1) Switch meter to standby if using a Corning meter, otherwise refer to instruction manual.

(2) Measure the sample temperature and adjust the temperature compensation knob accordingly.

(3) Wash the pH electrode with de-ionized water and wipe off the excess water using Kim Wipes.

(4) Insert the electrode into the sample - do not touch the bottom of the sample container with the electrode tip.

(5) Turn control knob to "read", allow the instrument to stabilize and note the pH reading on the scale.

(6) Turn control knob to "standby" before removing the electrode.

(7) Rinse the electrode with a small quantity of de-ionized water and then place the electrode tip in a small quantity of de-ionized water. The electrode should never be allowed to dry completely. This precaution should be observed even when the pH meter is not used for some time.

(8) Always check the instrument manual as the operational procedure may differ for individual meters.

## 3. LIGHT PENETRATION

### (a) Secchi Disc

Equipment

- Secchi disc and rope marked off in meters.

The secchi disc should be lowered into the water from the shaded side of the vessel until the outline of the disc is just perceptible, and the depth in meters noted. The lowering should be continued until it is completely out of sight and the depth noted: then the disc should be raised until the outline reappears, and this depth should also be noted. These two depths are then averaged and the result recorded.

For secchi colour refer to the code sheet and insert the combination of numerals which best describe the colour of the water as reflected on the disc. Many colours may be used to describe the colour of the water as reflected on the disc. Put the prevalent colour second, i.e. there is a difference between green-grey and grey-green.

## 4. BOTTOM SEDIMENT SAMPLES

Samplers - Ponar, Peterson, Ekman

Generally, the Ponar dredge is the most satisfactory device for the collection of all types of bottom samples.

The Ponar or Peterson dredge is used for collecting unconsolidated gravel, sand, silt, clay or marl samples.

The Ekman dredge is used for collecting samples from very soft bottoms such as mud, ooze and fine peaty materials.

### Operation of Dredges

(1) The designated dredge is lowered within 5 to 10 feet at which point it is allowed to free fall. When the Ekman dredge is used, a messenger must be sent down to trigger the jaws before retrieval.

(2) Open the dredge over a pail or pan in order to avoid loss of sample.

(3) Obtain a representative sample in accordance with the analyses on information to be obtained.

(4) The sample should be placed in a suitable water-tight container and labelled.

(5) Write a description of the bottom sample to include the texture, colour, odour, presence of organic matter.

# III CHEMICAL

## I DISSOLVED OXYGEN

### Modified Winkler Method

#### Equipment

1. Water sampler such as Nansen bottle - Kemmerer sampler, etc.
2. French bottle
3. 2 pipettes or equivalent, i.e. eye droppers that measure 2 ml aliquots
4. Automatic burette - reads up to 20 ml.
5. Prepared Re-agents:
   (i) Manganous sulfate
   (ii) Alkaline azide
   (iii) Sulfuric acid, 36N
   (iv) Sodium thiosulphate, 0.0125N
   (v) Starch

   Ensure that a fresh supply of re-agents are kept onboard.
6. A 1 litre volumetric flask.
7. A 100 ml. volumetric pipette (with large tip).
8. Erlenmeyer flask 250 ml.
9. Distilled water.

#### Obtaining Sample

Rinse bottle twice with sample water. The sample should be collected in such a way that a minimum of mixing with water and air occurs.

When taking a water sample from a Nansen bottle or Kemmerer sampler, allow approximately 25 mls to pass through the plastic tube rapidly so that air bubbles caught in the tube will be eliminated. Then tilt the French bottle so that one corner of the bottom is very low. Place the plastic tube in the lowest corner and allow the water to pass through it slowly. When the bottle is 1/4 full, the bottle may be filled at a faster rate. Allow the bottle to overflow and then take the tube from the French bottle keeping the water running until the tube is just about to break the surface. At this point, the sample flow should be stopped and the tube removed from the French bottle.

Chemical Analysis

Fixing the DO Sample

The water sample in the French bottle must be fixed immediately To fix the sample, add two (2) mls of manganous sulfate and two (2) mls of alkaline azide below the surface. Place the top on the French bottle so that no bubbles are trapped below the stopper. At this point, mix the sample by inverting the bottle several times. The floc which forms is generally brownish in colour. The formation of a thin white floc is indicative of a low DO.

Allow the bottle to stand quiescent until the flow has partially settled and then carefully remove stopper and add 2 mls of concentrated sulfuric acid down the neck of the bottle. Release stopper and shake

the bottle until a clear amber colour has developed. The sample is now ready for titration.

Titrating the Sample

Rinse a 250 ml Erlenmeyer flask with distilled water only (NOT WITH THE SAMPLE) and a 100 ml volumetric pipette with a small amount of the fixed sample. Pipette 100 ml of the sample to the flask.

Ensuring that the burette is zeroed (the bottom of the miniscus should always be read), titrate the sample with 0.0125N sodium thiosulphate until it becomes a pale straw colour.

Add 1-2 ml of starch solution which will cause the solution to turn a dark blue. Continue titrating until the blue colour just disappears. Swirl the flask continuously while titrating. The concentration of DO in ppm is read directly from the burette, i.e. 10.3 mls of titrant is equivalent to 10.3 ppm, provided that no correction is necessary due to an incorrect normality determined by standardization of the thiosulphate solution. Hence, if no correction is required, then 1 ml of titrant = 1 ppm DO.

Precautions:

1. Do not allow bubbles to enter the sample before it is fixed.
2. Do not rinse the Erlenmeyer flask with the sample.

3. The sample must be fixed as soon as it is taken.

4. If the sample is going to be left for any length of time before it is titrated, keep it away from direct sunlight.

## 2. ALKALINITY

### Procedures

### A - pH Meter Method

#### Equipment

- pH meter
- electric stirrer
- 250 ml beaker
- .02 N $H_2SO_4$
- automatic burette
- 100 ml volumetric pipette

Pipette 100 ml of the sample into the beaker which has previously been rinsed with a small amount of sample. Put the magnet into beaker, then start the stirrer. Put pH probes into sample making sure that the magnet does not hit the probes. Turn on pH meter and start to titrate slowly, then stop titrating and let the pH catch up and then start titrating

slowly again. This step should be followed as many times as is necessary especially near the end point which is a pH of 4.5. See also section III on pH.

1 ml of titrant = 10 mg/l of alkalinity measured as $CaCO_3$.

B - Methyl Orange Indicator End Point

Equipment

- automatic burette
- 0.2 N $H_2SO_4$
- 250 Erlenmeyer flask
- 100 ml volumetric pipette
- methyl orange indicator

Pipette 100 ml of the sample into 250 ml Erlenmeyer flask and add 2-3 drops of methyl orange indicator and titrate to end point. Obtain a buffer of pH 4.5 and add the indicator, this would be the colour of the end point. The sample is to be titrated to this colour and then 1 ml titrant = 10 mg/l alkalinity measured as $CaCO_3$.

3. CHLOROPHYLL

The amount of chlorophyll present in the water is an estimation of the standing crop of algae. For practical purposes this represents not only the standing crop but also the physiological activity in terms of photosynthesis. The type of chlorophyll present is also an indicator

of the type of algae present. Coccoid and filementous green algae contain chlorophyll a and chlorophyll b, while diatoms and brown flagellates contain a and c but lack chlorophyll b. Blue green algae contain chlorophyll a.

Methods of Sampling

A - by Chlorophyll Sampler

B - by Composite Samples

A - CHLOROPHYLL SAMPLER METHOD

All samples taken with the chlorophyll sampler are to be taken at 10 meters or at 1.5 meters from the bottom if the depth is less than 10 meters. The hose should be marked off into one meter units beginning at the intake end. Before lowering the hose into the water, open the valve at the hand pump. When the intake hose is at the predetermined depth, close the valve and pump the water into the precalibrated bucket. Fill the bucket to that mark which corresponds to the sampling depth level. Then open the valve and reel in the hose. The valve must be left open when the hose is lowered to allow the escape of air above the water column. Before pumping begins, the valve is closed. After the sample is collected open the valve to allow the hose to drain. It is important that this procedure be followed or an incorrect sample will be obtained.

Take a 250 ml aliquot from the bucket and filter using the millipore filter, if the 250 ml aliquot goes through quickly put in another 250 ml aliquot until the water goes through the filter very slowly or until 1,000 ml has passed through the filter. Always try to put multiples of 250 through the filter. Then put the filter paper into a small petri dish and label with the following information:

1. date
2. amount of sample filtered
3. sample number

and then store in cooler. If a millipore filter is unavailable, the sample is to be stored in a cool place and subsequently shipped to the lab.

Calibrating the Bucket

The bucket is marked in meter intervals up to 10 meters. Each line on the bucket is equivalent to the volume of water in one meter of the hose.

To calibrate the bucket various methods may be used. A meter section of hose may be used to fill the bucket and the appropriate levels marked onto the side of the bucket. If a meter section of hose is not available, calculate the volume of water in the hose by means of the following formula:

$$\pi \frac{d^2}{4} L = V$$

where d = diameter of the hose (cm)

L = length of hose (cm)

V = volume in ml

$\pi$ = 3.146

B - COMPOSITE SAMPLES METHOD

The composite sampling method is used if no chlorophyll sampler is available. In this method, a sample is taken at two depths by means of a Kemmerer sampler or a Nansen bottle. The surface sample is taken at 1.5 m and the bottom sample at 10 m, or if the total depth is less than 10 m at 1.5 m from the bottom. Equal amounts of water from each depth are mixed to form a composite sample which is filtered as described in the previous section. The amount from each depth is usually 500 ml but should be made considerably less in very turbid water.

If a millipore filter is unavailable, the sample is to be stored in a cool place and subsequently shipped to the lab.

# IV BIOLOGICAL

## I PHYTOPLANKTON

The phytoplankton sample is obtained in the same manner as the chlorophyll sample (composite or sampler - see previous section). However, instead of filtering, a sample is obtained in a 40 oz bottle to which 32 ml of formaldehyde is added as a preservative.

The bottle should be labelled as follows:

1. Phytoplankton
2. Sample number
3. Date
4. Station number
5. Body of water

## 2. ZOOPLANKTON

The vertical tow plankton net is a conical shaped No. 20 mesh nylon bolting cloth net. Attached to the bottom is a small metal bucket with openings covered with the same fine mesh netting. The net is lowered to a seven meter depth and then raised to the surface at a constant speed. When the net is brought up, the plankton trapped on the inside of the surface is washed down into the bucket using the tube on the outside of the net. The plankton is then drained from the bucket and preserved in a 4 oz size glass jar with sufficient formalin to effect a three per cent solution. The jar must be marked with

assigned sample number and the corresponding laboratory determination requested on the deck sheet.

3. BENTHIC FAUNA

Benthic fauna refers to those animal organisms found in the bottom sediments. The benthic population inhabiting any particular area is dependent upon many factors -- the characteristics of the substratum, the quality of the water, and certain physical features such as morphometry, currents, wave action, temperature, light and depth.

Sampling methods for benthic fauna are quantitative whenever possible. In rocky and in shallow areas, qualitative methods are employed.

Quantitative bottom samples for benthic fauna are collected with various sampling devices. The Ponar (similar to Peterson) dredge will be used for collecting samples from hard bottoms, such as sand, gravel, marl, clay and similar materials. This dredge is constructed of iron and is built so that its own weight and the leverage exerted by its closing mechanism bites its way into hard bottoms deeply enough to secure satisfactory samples.

The Ekman dredge is used for collecting samples from soft bottoms such as mud, ooze, and fine peaty materials. This dredge

consists of a square box of sheet brass the lower opening of which is closed by a pair of strong jaws. Two strong external springs, when released by a messenger, snap the jaws shut.

Another cast should be made if the first sample collected is questionable. The dredgings are washed in the field through a U.S. standard No. 25 mesh sleeve. The material retained on the sieve is transferred to 1 oz glass jars and preserved with 95% ethyl alcohol to fill container. Samples with are too large for these jars or not sieved in the field are placed in 4 oz or larger glass containers. The assigned sample number must be entered on the deck sheet and marked on the sample container.

# V RADIOLOGICAL

## IV RADIOLOGICAL

### Equipment

- half gallon plastic bottle
- waxed paper cups

### Procedure

Samples are collected in half gallon plastic bottles. The bottles are filled either by direct immersion or by means of waxed paper cups. If paper cups are used, a separate cup should be used for each sample and then discarded. Samples are normally taken only at the surface.

Bottles are marked with date and station number, and are forwarded collect to:

Protection Laboratory,
Ontario Department of Health,
360 Christie St.,
Toronto, Ontario.

Attn: Mr. E. Kennedy

Sample numbers are not required, but a record of how many and the location of samples collected should be sent to the office.

# VI FLOATING WASTE

1. OIL

Read this section thoroughly before starting survey of oil slick.

In the event of sighting an oil slick or when an oil slick is reported, the crew chief shall follow the procedure outlined below:

1. Suspend normal survey operations.
2. Inform the office immediately either by radio, telephone, or wire. Contact Mr. J. Kinkead, Mr. N. Vanderkooy, or Mr. J. Thon.
3. Determine the chart location of the oil spill and locate exact sampling points on the chart.
4. Attempt to locate the source.
5. Obtain statements from any eye witnesses together with their names and addresses.
6. Obtain at least a 5 ml sample of the oil for infrared analysis at the Toronto laboratory.
7. Complete request forms for sample analysis note: use proper OWRC "Pollution Complaint Form".
8. Place the samples in the special wooden box, lock the box and store in a safe place.
9. Inform the office of the action taken.
10. If the source or probable source is located, sample the outfalls and also take a sample at the industry or suspected source,

i.e. oil tanks if in the area. If you are going to go into the suspected source, check OWRC legal aspects of sampling in this book. (Appendix 1)

11. The wooden box is to be personally delivered to the driver, who must provide a signed receipt which should have the number of the box written on it.

12. Complete a field report together with charts to the office.

13. This procedure is to be followed when samples are taken from other abnormal discharges. The crew chief should bear in mind that court proceedings may result as a consequence of the sample data and follow procedures outlined in "Legal Aspects of Sampling" Appendix I.

Format of the Field Report

(a) State time at which the oil spill was sighted.

(b) State time when the oil spill was sampled.

(c) State time when the office was informed.

(d) State time when witnesses were interviewed.

(e) State names and addresses of witnesses.

(f) State time when the source was located.

(g) State the location of the true source or probable source area.

(h) State the size of the polluted area.

(i) State rate and direction of movement.

(j) State type of oil if possible:

(i) light or heavy, e.g. Bunker C, diesel, fuel, etc.
(ii) noticeable odour - sweet or sour

(k) estimated thickness and no. of gallons/sq.mi. from chart below.

| Gallons of oil per Square Mi. of Surface | Approximate film Thickness, inches | Appearance |
|---|---|---|
| 25 | $1.5 \times 10^{-6}$ | Barely visible under most favourable light conditions. |
| 50 | $3.0 \times 10^{-6}$ | Visible as a silvery sheen on the surface of the water. |
| 100 | $6.0 \times 10^{-6}$ | First trace of colour may be observed. |
| 200 | $12.0 \times 10^{-6}$ | Bright bands of colour are visible. |
| 666 | $40 \times 10^{-6}$ | Colour begins to turn dull. |
| 1,332 | $80 \times 10^{-6}$ | Colours are much darker. |

(l) State the size of the spill in acres (about 70 $yds^2$) or sq. miles.

# APPENDIX I

## LEGAL ASPECTS OF CONDUCTING SURVEYS AND SAMPLING PROCEDURES

## VII LEGAL ASPECTS OF CONDUCTING SURVEYS AND SAMPLING PROCEDURES *

As a result of considerable contact with the Legal Branch, with respect to the proposed prosecution of a major manufacturing company, many areas were brought to light where considerable improvements could be made, and subsequent time saved, by following a few simple guidelines when conducting any survey or sampling program at an industrial complex. Most of these guidelines will appear to be obvious but, nevertheless, they are frequently not adhered to by personnel out in the field.

All sampling programs and surveys should be conducted in such a manner that the information obtained as a result of the sampling program or survey will be of use should any legal action be contemplated in the future. Bear in mind that for the purposes of legal evidence only facts which can be supported are of any direct use. In certain cases, where facts cannot be obtained, expert opinion will be of value but factual data, i.e., what one perceives through the senses, remains all important. The assumptions, inferences and belief of witnesses are of little value for legal evidence.

Therefore, with the above in mind, the following items should be considered by personnel when conducting a sampling program or survey.

1. Correct Name of Company

It is important that the correct name of the company, containing all

---

* This section was prepared by staff from the Division of Industrial Wastes

punctuation and abbreviations as used by the company, be used in all references to the company in report material, letters, etc., e.g., it is not correct to write "John Smith's" when the true name of the company is "John Smith Manufacturing Co., Limited". A little care is all that is required to ensure that the correct name appears wherever reference to the company is made. If the person conducting the survey does not know the correct name of the company, he can obtain it from the Provincial Secretary's Department, Company Searches Office.

2. Correct Name of Watercourse

The correct name of the watercourse to which the wastes are discharged is important. Quite often, local people have a name for a creek, lake, river, etc., which is not the official or legal name. The local Department of Lands and Forests can frequently assist in determining the correct name for a watercourse. If necessary, the correct name can be obtained from the Surveyor General of the Lands and Surveys Branch of the Department of Lands and Forests.

Where the watercourse concerned is a storm ditch or a small creek with no name, then a general description of the location of the creek, its direction of flow, etc., sufficient to enable it to be clearly identified, is required.

3. Establish Proof of Sewer Flow

Where a sewer (or sewers) discharge to a watercourse, the person conducting the survey or sampling program should visibly inspect any sewer outfalls. Where sewers are underground with submerged outfalls, then an authorized company official should be consulted as to the point of discharge. This should then be reported as "Mr. 'Y', Plant Manager (etc.) reported that the sewer from a particular location discharged wastes to the watercourse in question through an outfall located at a particular point". This is not the same as assuming that the sewer discharged at a particular point. It is important that an authorized company official be consulted as to the origin of the wastes, the connection to the point of discharge and the location of the point of discharge of any or all sewers, even in the case where it is apparently obvious. In court, if you are called as a witness, the defense counsel will immediately ask you how you know that this sewer discharges at this point or how you know that the material being discharged comes from the accused's premises. Your reply can then be, "Mr. So and So told me".

It is important that you consult an official whose general authority would include the giving of such information. To be safe, the manager in charge of a plant or his superior should be consulted.

If the wastes are conveyed to a watercourse directly by an open ditch or closed sewer line whose course can be visually traced, then this

tracing should be carried out. In this case, your visual evidence is sufficient.

Where a company official is uncertain or does not know, then it is imperative that the person conducting the survey or sampling program determine the connection between the origin of the wastes and the point of waste discharge by some physical means such as a dye test.

Remember, unless it can be definitely established that the wastes originate in a certain place, namely, the accused's premises, and do discharge at a certain point, the succeeding evidence will be of no value in court. Therefore, even in cases where it is obvious, due to say the size of the outfall pipe or the magnitude of the operation or where previous survey reports have stated as such, that the sewer originating in the accused's premises discharges at a particular location, it is still necessary to obtain adequate evidence of this.

4. Sampling

All sampling must be carried out by OWRC personnel or under the direct supervision of OWRC personnel, where the samples are taken manually. Here again, the implication is that you must be able to swear to the fact that you personally saw that the samples were taken on a particular day at a particular location.

In certain plants, where the company has automatic sampling equipment installed, it is common practice to use automatically composited

samples for analytical tests. This is acceptable providing that the sample bottles are filled by OWRC personnel or at least under the direct supervision of OWRC personnel and it is clearly understood by our personnel how the machine operates and they observe that it is operating to take the particular samples. It is useless, generally, to let company personnel obtain samples for you in your absence. Where possible, composite samples from company owned and operated equipment taken by our staff or under their supervision should be backed up with a manually composited sample of reasonable duration taken by OWRC personnel.

5. Samples

It is assumed that all the necessary precautions will be observed in obtaining samples and filling sample bottles.

Having accomplished this, it is necessary to label the sample bottles and complete a laboratory analytical requirement sheet - Form No. 72-0345-12699. The need for accuracy and consistency in these operations must be stressed. The correct name of the company, location, type of sample (grab or composite), sample description, time and date must all be accurate and consistent.

Terminology for the description of a sewer should be decided on, probably after consultation with the authorized company official, and maintained as such for all further references to that sewer. There have

been cases where during 24 hour continuous sampling, different descriptions have been used by people sampling on different shifts to describe the same sewer. This is not only confusing but involves a good deal of extra typing to correct the inconsistencies and requires re-certification by the laboratory staff. Care and co-operation in this area can save a great deal of future work.

It is desirable to take all steps that are available to ensure that the sample bottles are not tampered with prior to the analyses being made, i.e., sealing the bottles.

6. Analysis Requirements

Do not ask for analyses which you are not able to subsequently interpret or which have no real value. There have been instances where certain analyses were requested as routine and were subsequently found to be of no value to the particular samples. A little thought to decide whether analyses are necessary or can be interpreted meaningfully, before they are requested, can save considerable work in the laboratory, in typing, etc. Be sure all requirements of the laboratory analysts in order to certify the analyses are met, i.e., 6 hour time limit for bacteriological analysis.

7. Flow Data

Flow data should preferably be obtained in writing from the

company, if the company is being relied upon to provide this information. If this is impractical or not possible, then oral information is acceptable (and this also applies to obtaining written flow data from the company) providing that the name of the person who supplied the information, and the time and date, are recorded and the person who supplied it is authorized to give such information. It is also of benefit in this case to have a witness who can be called upon, if necessary, to substantiate the source of information.

If flow recorders are installed, the original charts should be obtained where possible, provided you obtain the name and address of the employee who operated the instrument that made the charts or whose job it was to obtain the charts from the instrument and who is familiar with its operation. Failing this, the recorder should be read directly by the person conducting the survey or sampling program. Additional information, such as conversion factors, etc., should be obtained by direct request to authorized company officials. In all cases of obtaining information from the company, it is necessary that it be obtained from an authorized source and that the name and address of the official also be obtained so that he can be subpoened as a witness.

Where an estimate of the flow is made because no flow measuring equipment is available, the person making the estimate should be prepared to indicate in detail the method used and the probable degree of accuracy.

8. General Information

Visual inspection of the receiving watercourse, both above and below plant outfalls, should be made to determine the existence of any sensory evidence of pollution; foam, colour, floating solids, debris, smells, etc. Coloured photographs or slides should be taken and they should be identified by the taker by his signature, the date of taking and the location and object taken.

Contact local departments such as Lands and Forests, Health Unit, etc., and discuss the problem and any possible harmful effects that can be followed up. Any evidence of odorous waters, tainting of fish, decline of fish population, harm to animal life or vegetation, and complaints from residents should be followed up. Names and addresses of people who relay this information should be noted and any contacts should be followed up where possible.

It is important to have as few OWRC personnel as possible take samples and otherwise obtain evidence. Some planning will ensure that the same personnel, where possible, will carry out the various matters related to the collection of evidence.

Ideally, the results of any survey or sampling program should be applicable for use in legal proceedings without further field work. Observance of the above, while not being able to guarantee "perfect evidence", will certainly, in general, improve the usefulness of the results from most surveys and sampling programs.

Note:

Staff from the Division of Industrial Wastes should be notified when sampling is carried out for legal purposes at an industry.

# APPENDIX III

## DISSOLVED OXYGEN

## CONVERSION TABLE

## TABLE I

## SATURATION VALUES OF DISSOLVED OXYGEN, IN PARTS PER MILLION, FOR TEMPERATURES IN °C. RANGING FROM 0°C. to 30°C.

(Under Normal Atmosphere at 760 mm. Pressure)

| Temp. (°C.) | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 14.62 | 14.58 | 14.54 | 14.50 | 14.46 | 14.42 | 14.39 | 14.35 | 14.31 | 14.27 |
| 1 | 14.23 | 14.19 | 14.15 | 14.11 | 14.07 | 14.03 | 14.00 | 13.96 | 13.92 | 13.88 |
| 2 | 13.84 | 13.80 | 13.77 | 13.73 | 13.70 | 13.66 | 13.62 | 13.59 | 13.55 | 13.52 |
| 3 | 13.48 | 13.44 | 13.41 | 13.38 | 13.34 | 13.30 | 13.27 | 13.24 | 13.20 | 13.16 |
| 4 | 13.13 | 13.10 | 13.06 | 13.03 | 13.00 | 12.97 | 12.93 | 12.90 | 12.87 | 12.83 |
| 5 | 12.80 | 12.77 | 12.74 | 12.70 | 12.67 | 12.64 | 12.61 | 12.58 | 12.54 | 12.51 |
| 6 | 12.48 | 12.45 | 12.42 | 12.39 | 12.36 | 12.32 | 12.29 | 12.26 | 12.23 | 12.20 |
| 7 | 12.17 | 12.14 | 12.11 | 12.08 | 12.05 | 12.02 | 11.99 | 11.96 | 11.93 | 11.90 |
| 8 | 11.87 | 11.84 | 11.81 | 11.79 | 11.76 | 11.73 | 11.70 | 11.67 | 11.65 | 11.62 |
| 9 | 11.59 | 11.56 | 11.54 | 11.51 | 11.49 | 11.46 | 11.43 | 11.41 | 11.38 | 11.36 |
| 10 | 11.33 | 11.31 | 11.28 | 11.25 | 11.23 | 11.21 | 11.18 | 11.15 | 11.13 | 11.11 |
| 11 | 11.08 | 11.06 | 11.03 | 11.00 | 10.98 | 10.96 | 10.93 | 10.90 | 10.88 | 10.86 |
| 12 | 10.83 | 10.81 | 10.78 | 10.76 | 10.74 | 10.71 | 10.69 | 10.67 | 10.65 | 10.62 |
| 13 | 10.60 | 10.58 | 10.55 | 10.53 | 10.51 | 10.48 | 10.46 | 10.44 | 10.42 | 10.39 |
| 14 | 10.37 | 10.35 | 10.33 | 10.30 | 10.28 | 10.26 | 10.24 | 10.22 | 10.19 | 10.17 |
| 15 | 10.15 | 10.13 | 10.11 | 10.09 | 10.07 | 10.05 | 10.03 | 10.01 | 9.99 | 9.97 |
| 16 | 9.95 | 9.93 | 9.91 | 9.89 | 9.87 | 9.85 | 9.82 | 9.80 | 9.78 | 9.76 |
| 17 | 9.74 | 9.72 | 9.70 | 9.68 | 9.66 | 9.64 | 9.62 | 9.60 | 9.58 | 9.56 |
| 18 | 9.54 | 9.52 | 9.50 | 9.48 | 9.46 | 9.44 | 9.43 | 9.41 | 9.39 | 9.37 |
| 19 | 9.35 | 9.33 | 9.31 | 9.30 | 9.28 | 9.26 | 9.24 | 9.22 | 9.21 | 9.19 |
| 20 | 9.17 | 9.15 | 9.13 | 9.12 | 9.10 | 9.08 | 9.06 | 9.04 | 9.03 | 9.01 |
| 21 | 8.99 | 8.98 | 8.96 | 8.94 | 8.93 | 8.91 | 8.89 | 8.88 | 8.86 | 8.85 |
| 22 | 8.83 | 8.81 | 8.80 | 8.78 | 8.77 | 8.75 | 8.74 | 8.72 | 8.71 | 8.69 |
| 23 | 8.68 | 8.66 | 8.65 | 8.63 | 8.62 | 8.60 | 8.59 | 8.57 | 8.56 | 8.54 |
| 24 | 8.53 | 8.51 | 8.50 | 8.48 | 8.47 | 8.45 | 8.44 | 8.42 | 8.41 | 8.39 |
| 25 | 8.38 | 8.36 | 8.35 | 8.33 | 8.32 | 8.30 | 8.28 | 8.27 | 8.25 | 8.24 |
| 26 | 8.22 | 8.20 | 8.19 | 8.17 | 8.16 | 8.14 | 8.13 | 8.11 | 8.10 | 8.08 |
| 27 | 8.07 | 8.05 | 8.04 | 8.02 | 8.01 | 7.99 | 7.98 | 7.96 | 7.95 | 7.93 |
| 28 | 7.92 | 7.90 | 7.89 | 7.87 | 7.86 | 7.84 | 7.83 | 7.81 | 7.80 | 7.78 |
| 29 | 7.77 | 7.75 | 7.74 | 7.73 | 7.71 | 7.70 | 7.69 | 7.67 | 7.66 | 7.64 |
| 30 | 7.63 | | | | | | | | | |